#308

Micro- and macro- elastic properties of tungsten fiber-reinforced tungsten composites probed by nano-indentation and laser ultrasonics

S. Ando, H.T. Lee*, J.W. Coenen[a], Y. Mao[a], R. Kasada[b], J. Riesch[c], and Y. Ueda

Graduate School of Engineering, Osaka University, Suita, Osaka 565-0871, Japan

[a] Forschungszentrum Jülich GmbH, Institut für Energie- und Klimaforschung – Plasmaphysik, Partner of the Trilateral Euregio Cluster (TEC), 52425 Jülich, Germany

[b] Institute of Advanced Energy, Kyoto University, Uji, Kyoto 611-0011, Japan

[c] Max-Planck-Institut für Plasmaphysik, 85748 Garching, Germany

Presenter email : ando-s@st.eie.eng.osaka-u.ac.jp * Corresponding author email : heunlee@eei.eng.osaka-u.ac.jp

大阪大学 OSAKA UNIVERSITY

Introduction

Tungsten fiber-reinforced tungsten composites (W_f/W) are presently being developed in the EU as next generation W materials for plasma-facing components. They possess pseudo-ductility and can overcome some of the limitations caused by the inherent brittleness of pure W. Material properties must be well characterized to allow detailed component design and analysis.

The macroscopic mechanical properties of composites depend critically on the microscopic interplay of the **matrix**, **interface**, and **fiber**.

If W_f/W composites are to be used as plasma-facing materials, the effect of hydrogen plasma exposure on the mechanical properties needs to be clarified.

Purpose

To characterize elastic properties of W_f/W by elucidating the interaction of W-matrix and W-fiber and to investigate the effects of hydrogen inclusion on mechanical properties

Conclusion

◆**Summary:** The purpose of this research was to determine the effect of fiber volume fraction on the elastic properties of W_f/W samples manufactured using SPS technique. Laser ultrasonic measurements indicate change in bulk modulus between 20-40% fiber volume fraction, while no significant difference was observed between 40-60% fiber volume fraction. To elucidate the dominant contribution on the micro-scale, nano-indentation measurements were performed to measure the strength of fiber and matrix separately.

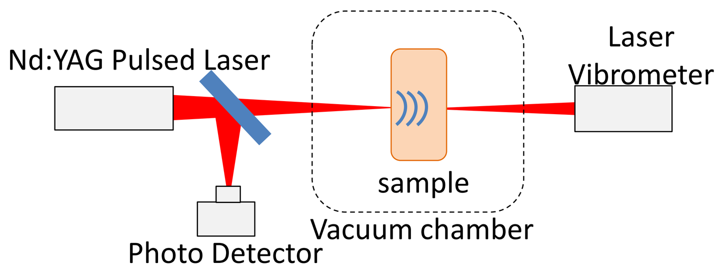
◆**Conclusions**

- Using a simple mixture model considering fiber shortness, we find good agreement between laser ultrasonics and nano-indentation measurements – suggesting bulk properties can be described/estimated by linear superposition of fiber and matrix strengths scaled by the fiber volume fraction.
- Using the present synthesis method, 40~50% fiber volume fraction is highest Young's modulus due to poorer matrix properties at higher fiber volume fraction. Improved synthesis method to increase the matrix strength or using longer fibers may provide a realistic path towards creating stronger W_f/W composite materials approaching pure-W properties in the elastic regime with the additional benefit of pseudo-ductility.

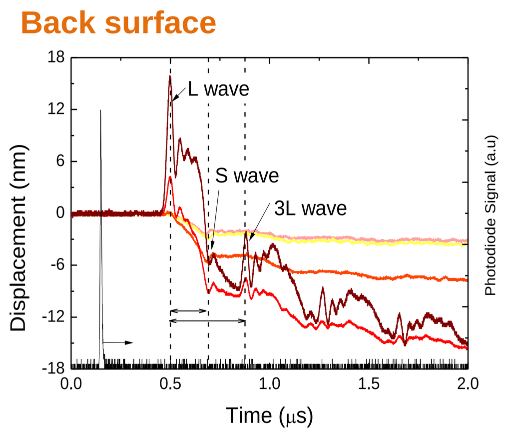
◆**Further work required:** The effect of D-irradiation on the elastic properties was within the scatter of the data.

Experimental setup

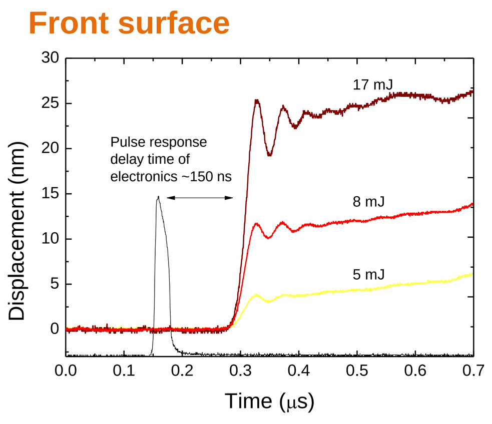
1) Laser ultrasonic method



Back surface



Front surface

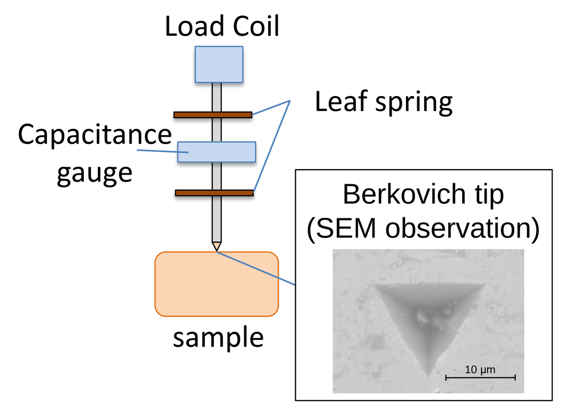


Excitation laser (Nd: YAG 1064 nm)
- Pulse FWHM ~15 ns
- Beam focused to ≤ 1 mm diameter

Polytec vibrometer
- He-Ne laser (632 nm)
- Surface displacement ±75 nm
- Surface velocity up to 3 m/s
- Bandwidth up to 24 Mhz

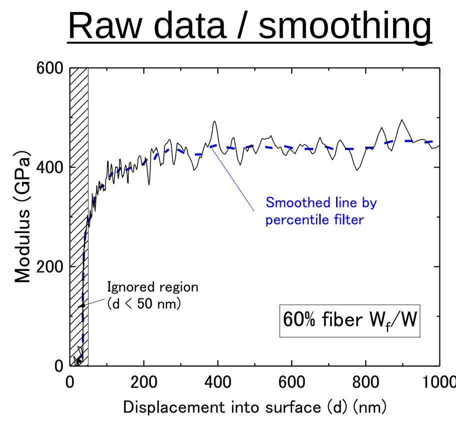
- Increasing excitation laser power results in shift from thermoelastic to ablation regime
- Longitudinal and shear wave velocities are determined from time of flight
- Propagating distance are determined from sample thickness measured by micro-meter
- Propagating time are measured by delay time of first and reflected wave

2) Nano-indentation method

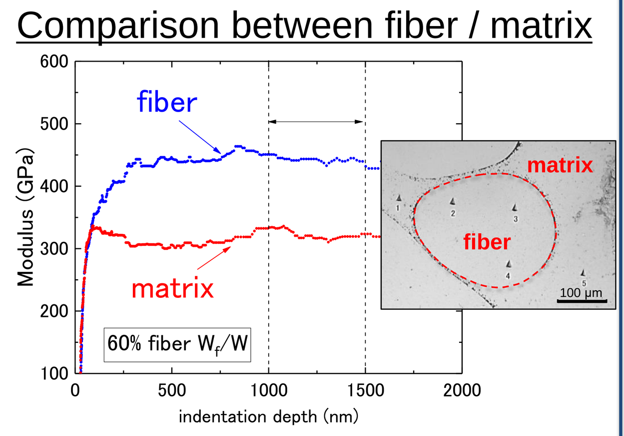


- Agilent Tech, Nano Indentor G200
- CSM mode
- Depth-dependent hardness/modulus measurements

Raw data / smoothing

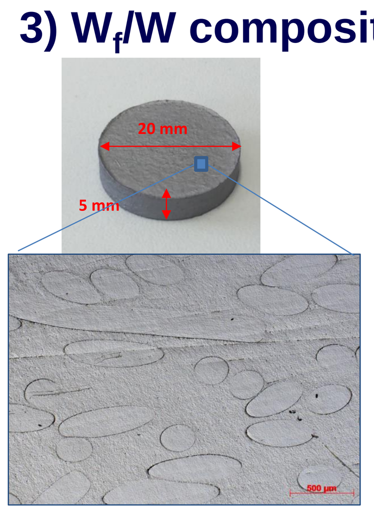


Comparison between fiber / matrix



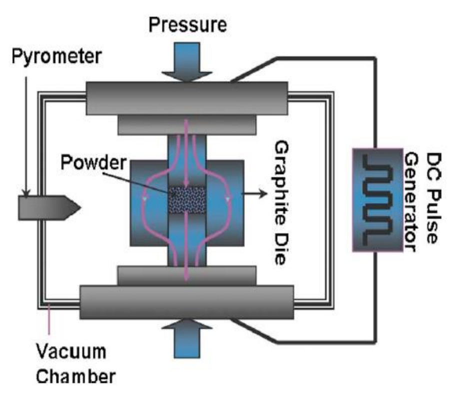
- Matrix of 3 by 4 points or line scan of 15 points were taken with 100 μm separation
- The hardness or modulus data between 1000-1500 nm was averaged to avoid "surface" effects

3) W_f/W composite samples



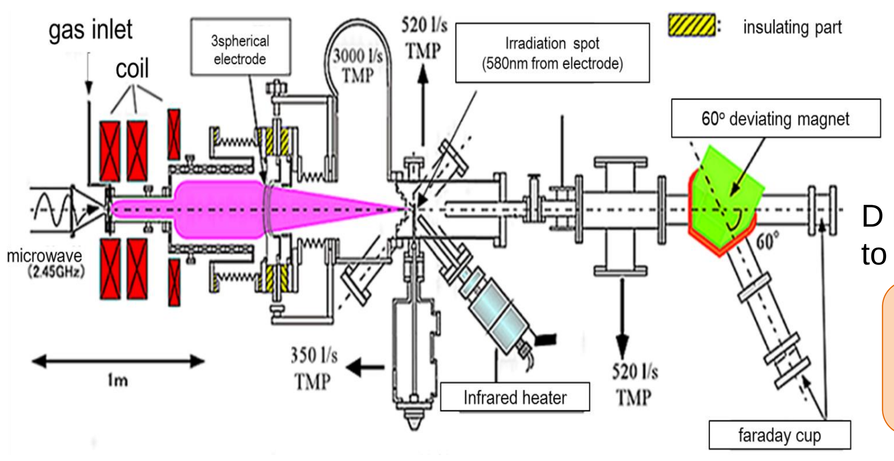
- W powder grain: 5 μm
- W-fiber length: 2.4mm
- W-fiber diameter: 0.24 mm
- Multi fibers / random distribution
- W-fiber volume fraction: 20% - 60%
- Compared to reference W from ALMT Corp, Japan

Method: Spark Plasma Sintering (SPS)



- Pulsed DC current directly passes through and heats the graphite die
- High heating or cooling rate enables to sinter metal composites

4) D irradiation experiments



D irradiation conditions

Incident flux : $1.0 \times 10^{20}\ m^{-2}s^{-1}$

V_{acc} : $1\ keV$

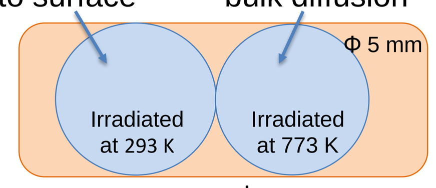
Time : $1800\ s$

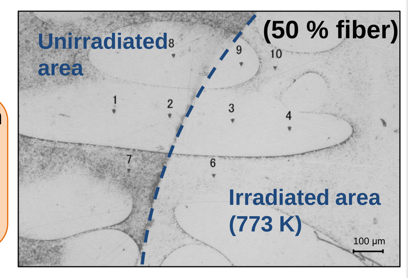
Fluence : $1.8 \times 10^{23}\ m^{-2}$

Irradiation temp : 20℃ (293 K), 500℃ (773 K)

D more limited to surface

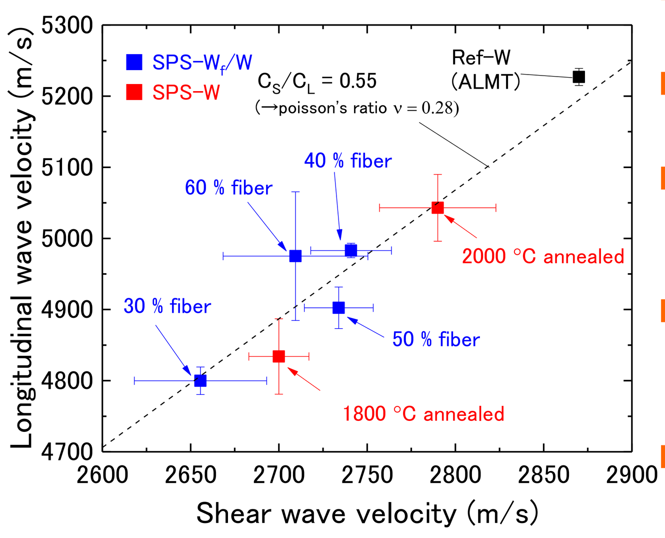
Enhanced D bulk diffusion





Results & Discussion

1) Longitudinal and shear wave velocities from laser ultrasonic



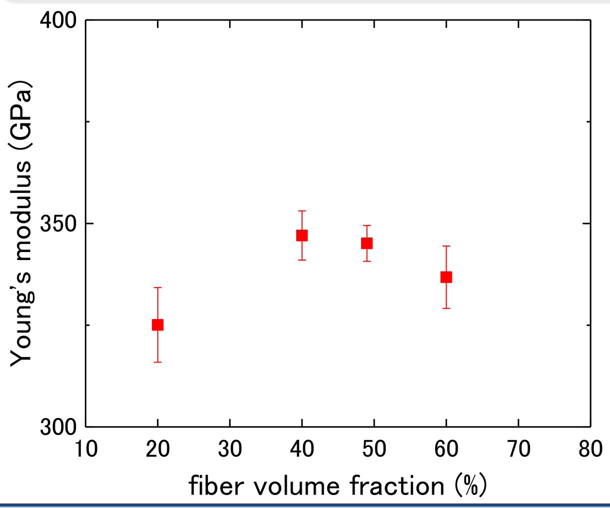
- Data for all 9 measurement spots were averaged
- Wave velocities do not scale with increasing fiber volume fraction
- Wave velocity differences between pure W samples arise due to density (porosity)
- Poisson's ratio is expressed as a function of C_L/C_S

$$\nu = \frac{1-(C_L/C_S)^2}{2\{(C_L/C_S)^2-1\}}$$

- Poisson's ratio of W_f/W are similar to pure W samples → indicates W_f/W samples can be treated as isotropic at macro level

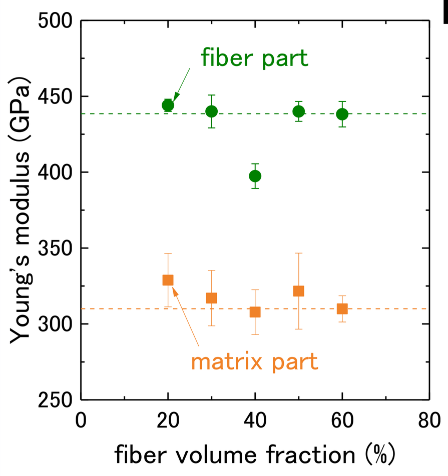
◆Relationship between wave velocities and elastic constants for isotropic material

$$E = \rho c_S^2 \frac{3c_L^2 - 4c_S^2}{c_L^2 - c_S^2} \quad \left\{ \begin{array}{ll} E : Young's\ modulus & c_L : L\ wave\ velocity \\ \rho : Density & c_S : S\ wave\ velocity \end{array} \right\}$$



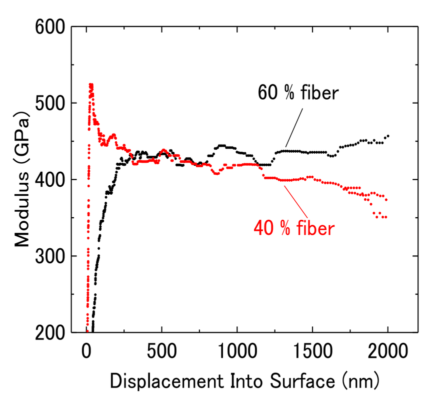
- Laser ultrasonics measurements probes the integrated properties along the transport path.
- Modulus improvement by fiber are observed between 20 – 40 % fiber volume fraction
- No significant difference between 40 – 60 % fiber volume fraction

2) Matrix and fiber modulus from nano-indentation



Fiber
- 440 GPa except for 40% sample
- 40 GPa lower for 40 % fiber sample
- Reasons for this discrepancy is unclear – Raw data qualitatively different

Matrix
- 310 GPa at all samples
- Standard deviation by variation of measurement spot are 15 GPa



- Suggests fiber surface properties are different
- Averaged data for all 10 measurement spots at fiber or matrix in each samples is shown
- Each measurement spot was averaged at a depth from 1 μm to 1.5 μm

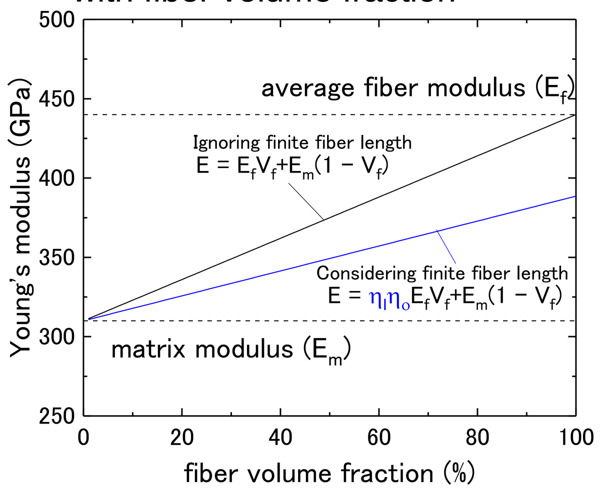
◆Prediction of Young's Modulus in short-fiber composite [1]

$$E_{comp} = \eta_l \eta_o E_f V_f + E_m(1 - V_f)$$

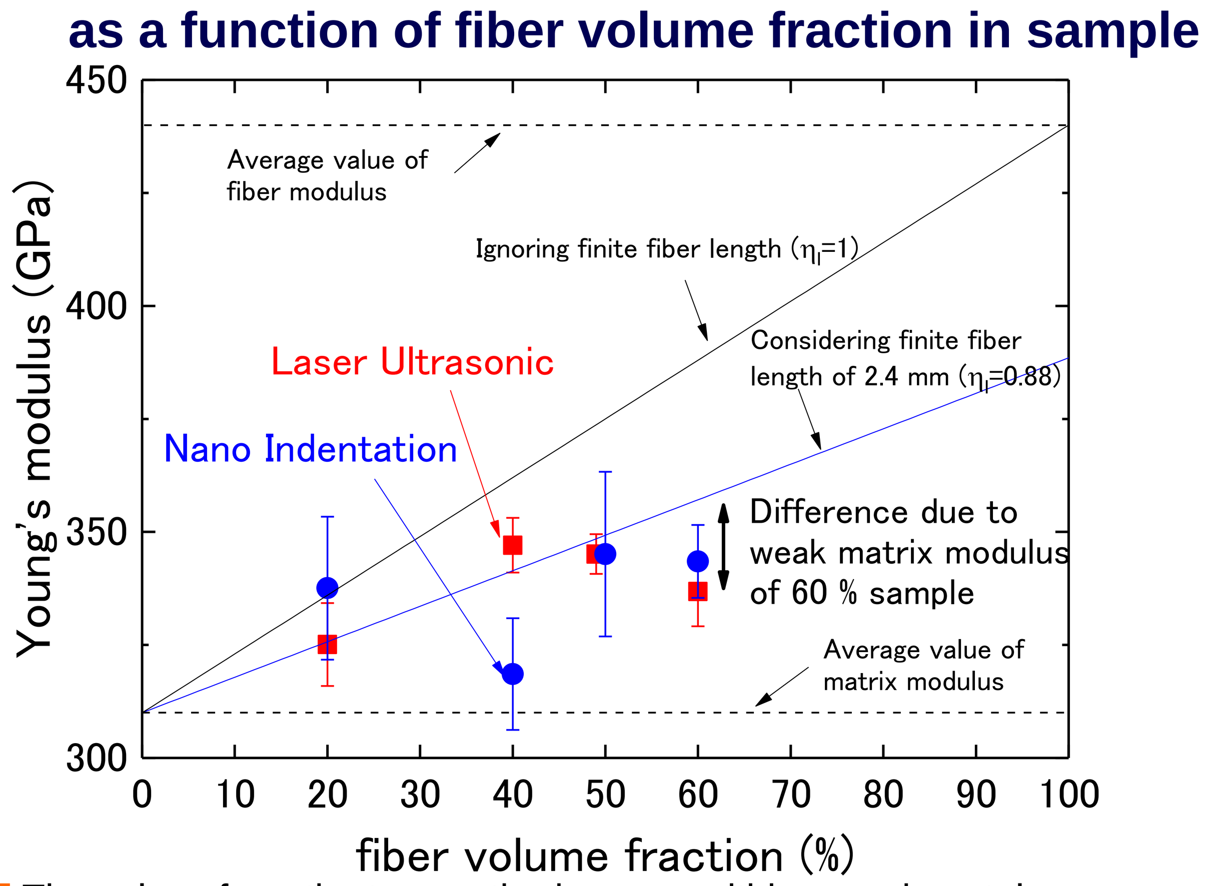
V_f : volume ratio of fiber
E_f (= 430 GPa) : fiber modulus
E_m (= 310 GPa) : matrix modulus
η_l (= 0.88) : Stiffness reduction ratio due to shortness of fiber
η_o (= 1) : Stiffness reduction ratio due to fiber orientation

[1] Yukio SANOMURA, "Prediction of Young's Modulus and Strength of Extrudate in Short-Fiber-Reinforced Polypropylene"

Simple mixture model suggests strength monotonically increases with fiber volume fraction



3) Comparison of modulus by two methods as a function of fiber volume fraction in sample

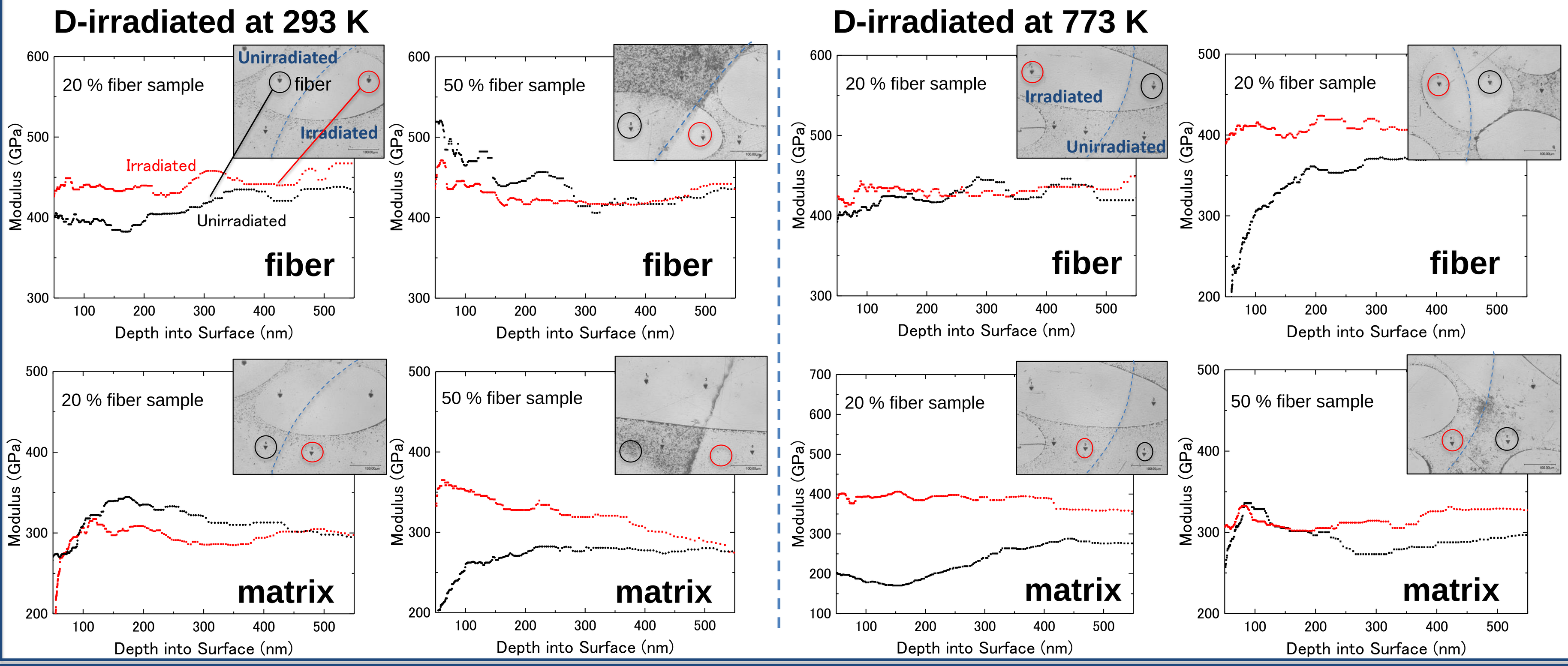


- The values from the two methods agree within experimental error except for 40 % fiber volume fraction sample – suggests simple mixture model is sufficient to characterize bulk properties
- Monotonic increase in strength was not observed for 60 % fiber volume fraction sample – suggests that optimum levels for present synthesis method is between 40-50% fiber volume fraction
- Improved bulk elastic properties of W_f/W composite samples may be possible by increasing matrix strength or by using longer fibers

4) Deuterium irradiation effect on elastic properties of W_f/W composite samples

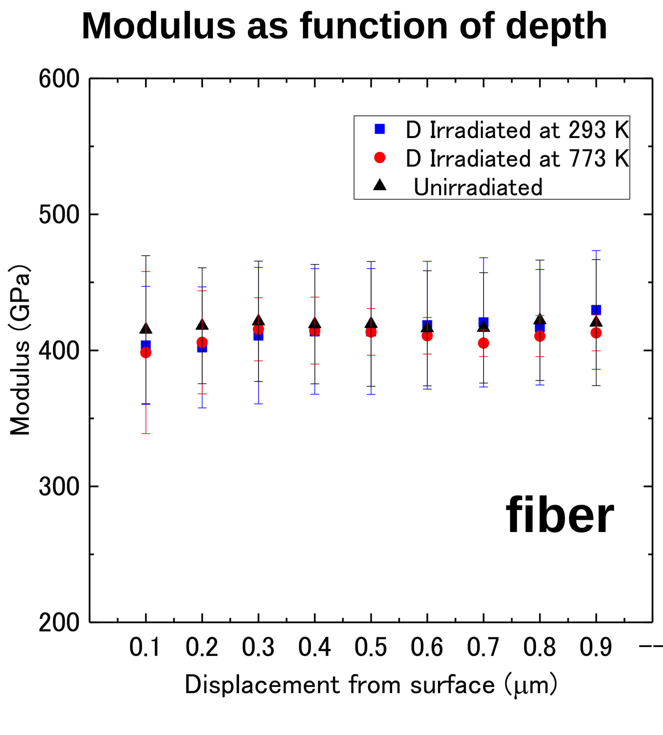
D-irradiated at 293 K

D-irradiated at 773 K



- There is great variability/scatter in the material properties of a single sample depending on location
- To quantify the effects of D irradiation, an attempt was made to examine relative changes in the modulus in a local region separating the irradiated and unirradiated areas (both fiber and matrix)
- Representative data of modulus determined from nano-indentation is shown
- Irradiated and unirradiated areas correspond to red and black lines, respectively
- D-irradiation could result in an increase or decrease or no changes in the hardness/ modulus measurements for both fiber and matrix

Modulus as function of depth



- Averaged values of modulus as a function of depth are shown for unirradiated and D-irradiated samples (293 K, 773 K)
- The error bar shows the range of scatter between 10 measurement spots
- Effect of D is within the scatter of the data